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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

*Space Programs Summary No. 37-33, Volume V*

for the period April 1, 1965, to May 31, 1965

## Supporting Research and Advanced Development

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**JET PROPULSION LABORATORY**  
**CALIFORNIA INSTITUTE OF TECHNOLOGY**  
**PASADENA, CALIFORNIA**

June 30, 1965

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## Preface

The *Space Programs Summary* is a six-volume, bimonthly publication that documents the current project activities and supporting research and advanced development efforts conducted or managed by JPL for the NASA space exploration programs. The titles of all volumes of the *Space Programs Summary* are:

- Vol. I. The Lunar Program (Confidential)
- Vol. II. The Planetary-Interplanetary Program (Confidential)
- Vol. III. The Deep Space Network (Unclassified)
- Vol. IV. Supporting Research and Advanced Development (Unclassified)
- Vol. V. Supporting Research and Advanced Development (Confidential)
- Vol. VI. Space Exploration Programs and Space Sciences (Unclassified)

The *Space Programs Summary*, Vol. VI consists of an unclassified digest of appropriate material from Vols. I, II, and III; an original presentation of technical supporting activities, including engineering development of environmental-test facilities, and quality assurance and reliability; and a reprint of the space science instrumentation studies of Vols. I and II.



W. H. Pickering, Director  
Jet Propulsion Laboratory

### Space Programs Summary No. 37-33, Volume V

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# PROPULSION DIVISION

## I. Solid Propellant Engineering

### A. High-Energy Propellant Development

F. A. Anderson

#### 1. Hydrazine Diperchlorate Characterization

Earlier studies of hydrazine diperchlorate as a potential high-energy oxidizer were reported in SPS 37-31 and in previous *Summaries*. During this reporting period, chemical analyses have been continued which included samples from a lot of hydrazine diperchlorate (HP<sub>2</sub>) obtained from the Thiokol Chemical Corporation. The study of the effects of moisture on the properties of HP<sub>2</sub> have also been continued. In addition, studies were made of the thermal stability and properties, crystallinity, moisture absorption rate at various relative humidities, and compatibility of HP<sub>2</sub> with binder ingredients. Some small-scale (5-g size) propellant mixing has been started.

#### 2. Chemical Analysis of HP<sub>2</sub>

Various samples from a lot of HP<sub>2</sub> obtained from the Thiokol Chemical Corporation have been analyzed and compared with the analyses of the two lots of HP<sub>2</sub> obtained from the Naval Propellant Plant, reported in SPS 37-31. Table 1 shows the comparative analysis of four

Table 1. Hydrazine diperchlorate analysis

Property	Theoretical value	NPP-1 <sup>c</sup> A	NPP-1 B	TCC-1 <sup>d</sup> 1	TCC-1 3
Hydrazine diperchlorate, %	100.0	96.16	97.21	95.85	96.33
Hydrazine monoperochlorate, %	0.0	2.41	0.65	2.22	3.26
Hydrazine content, %	13.73	13.77	13.40	13.18	13.10
Perchlorate content, %	85.41	83.97	—	83.50	85.73
Total chloride, %	30.47	30.19	30.26	29.82	30.12
Moisture content, %	—	—	—	0.82	0.69
Melting point <sup>a</sup> , °C	—	197	197	196	196
Density, gm/cc	—	2.25	2.25	2.16	2.20
Impact sensitivity <sup>b</sup> , in.-lb	—	68	68	66	66

<sup>a</sup> Melting point determined in a Perkin-Elmer differential scanning calorimeter, Model DSC-1.

<sup>b</sup> 50% point as determined by Bruceton method.

<sup>c</sup> HP<sub>2</sub> from Naval Propellant Plant.

<sup>d</sup> HP<sub>2</sub> from Thiokol Chemical Corporation.

different samples. As can be noted, the difference in analyses between the two different source materials does not appear to be any greater than the difference between samples from the same lot of  $HP_2$ .

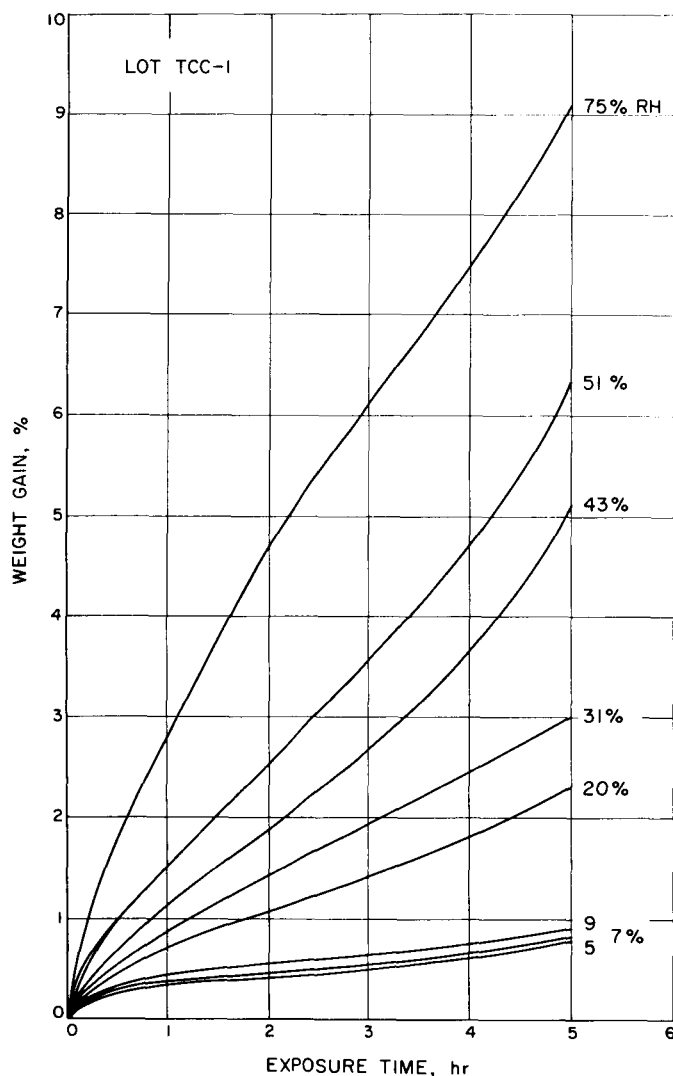


Fig. 1. Moisture absorption rate of  $HP_2$  at various relative humidities

### 3. Moisture Sensitivity of $HP_2$

As has been reported previously, hydrazine diperchlorate is extremely hygroscopic. An attempt is being made to determine what effects moisture has on the  $HP_2$  and its properties, and what the tolerable limits of relative humidity are for satisfactory handling of  $HP_2$ . An attempt is being made at this time to determine whether moisture causes decomposition of the  $HP_2$  or simply tends to cause hydration. Preliminary tests suggest a tendency of the  $HP_2$  to hydrate in the presence of moisture. On exposure to even low relative humidities, the moisture absorption rate is rather high initially, the rate decreasing with time. Coincident with the moisture absorption, a very rapid desensitization of the  $HP_2$  occurs, specifically a rapid decrease in the sensitivity to impact. If the presence of moisture caused decomposition to occur, then the byproducts of the decomposition would include hydrazine monoperchlorate and free perchloric acid. Either of these materials would be expected to make the  $HP_2$  more sensitive to impact. It is known that the monoperchlorate is considerably more sensitive than the diperchlorate. Fig. 1 shows the moisture absorption rate of  $HP_2$  at various relative humidities. As will be noted, the absorption rate is high initially but it also decreases rapidly at the lower relative humidities, 9%

Table 2. Effect of moisture on the impact sensitivity of hydrazine diperchlorate, Lot No. TCC-1

Relative humidity, %	Impact sensitivity in inches of drop height <sup>b</sup> after exposure times of				
	0 min	15 min	30 min	60 min	120 min
0	15	15	15	15	15
5	15	20.5	21.6	20.5	21.7
9	15	19.8	21.1	21.0	22.2
20	15	22.2	22.3	22.5	>48
35	15	22.0	24.0	>26	>48

<sup>a</sup> Based on average value of several tests run on different days.  
<sup>b</sup> Mean value based on a sample of 14 specimens minimum.

Table 3. Moisture absorption rate of  $HP_2$ , percentage weight gained per minute

Time, min	Weight gained at indicated relative humidity, % /min							
	5 %	7 %	9 %	20 %	31 %	43 %	51 %	75 %
15	0.0135	0.0143	0.0159	0.0206	0.0231	0.0299	0.0314	0.0467
30	0.0085	0.0096	0.0109	0.0150	0.0171	0.0224	0.0359	0.0461
60	0.0061	0.0065	0.0067	0.0121	0.0148	0.0193	0.0251	0.0472
120	0.0035	0.0039	0.0046	0.0090	0.0121	0.0161	0.0212	0.0396
300	0.0027	0.0028	0.0030	0.0078	0.0100	0.0170	0.0210	0.0345

or less. In a relative humidity of 9%, less than 1% weight gain was experienced after a 5-hr exposure. Approximately a 30% increase in drop-height with a 4-lb ball occurs within 15 min of exposure time even at relative humidities as low as 5%. This change remains relatively constant over a period of 2 hr exposure for relative humidities up through 9%. At 20% RH the decrease in impact sensitivity appears to be of the same order of magnitude for approximately 1 hr. After 2 hr at 20% RH, however, no detonations were experienced at a drop height of 48 in. with a 4-lb ball—the limit of the JPL impact tester. Table 2 shows the change in impact sensitivity as a function of exposure time to different relative humidities. Table 3 shows the change in the moisture absorption rate of  $\text{HP}_2$  at various relative humidities as a function of time.

#### 4. Thermal Stability of $\text{HP}_2$

A study has been made, and is continuing, of the thermal properties of hydrazine dperchlorate. A Perkin-

Elmer differential scanning calorimeter, Model DSC-1, is being used to determine the melting point and decomposition temperature and also to study the effects of moisture on the thermal properties of the  $\text{HP}_2$ .

The  $\text{HP}_2$  goes through a crystal phase change at approximately  $90^\circ\text{C}$ . Fig. 2 is a thermogram of a dry sample of  $\text{HP}_2$  and shows this phase change as the first endotherm. This phase change has also been observed and photographed through a hot stage microscope, using a polarized light source. Referring again to the thermogram (Fig. 2), the melting point is shown as an endotherm at approximately  $197^\circ\text{C}$  ( $470^\circ\text{K}$ ). The melting is followed by a sharp exotherm which peaks at approximately  $207^\circ\text{C}$ . The final exotherm and complete decomposition then occurs near  $300^\circ\text{C}$ . The evidence of what happens at the first epotherm is inconclusive at this time and is being studied further. One probability, however, is that 1 mole of perchloric acid is given up at this point, yielding the monoperchlorate salt. Fig. 3 is a thermogram of a sample of  $\text{HP}_2$  which had been conditioned in an

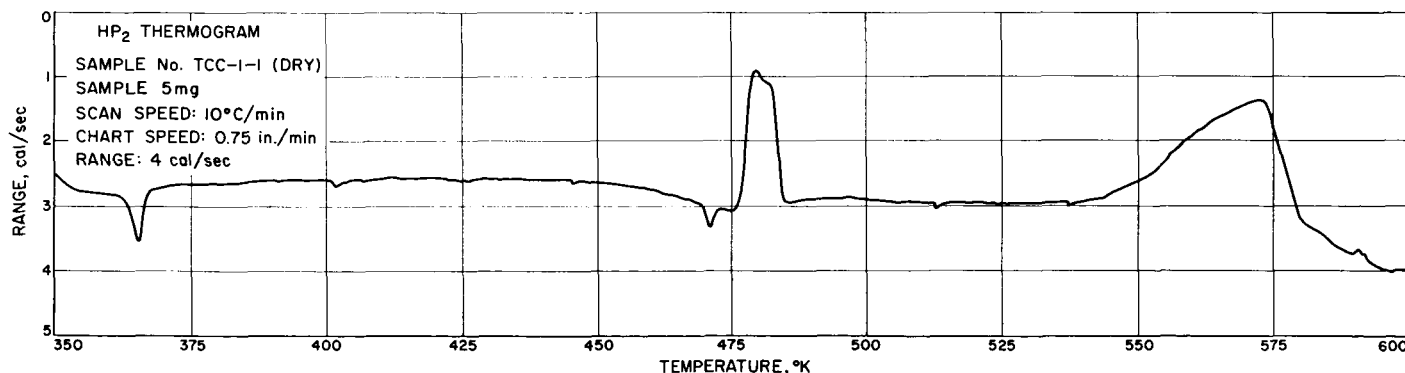


Fig. 2. Thermogram of dry  $\text{HP}_2$ , Sample TCC-1-1

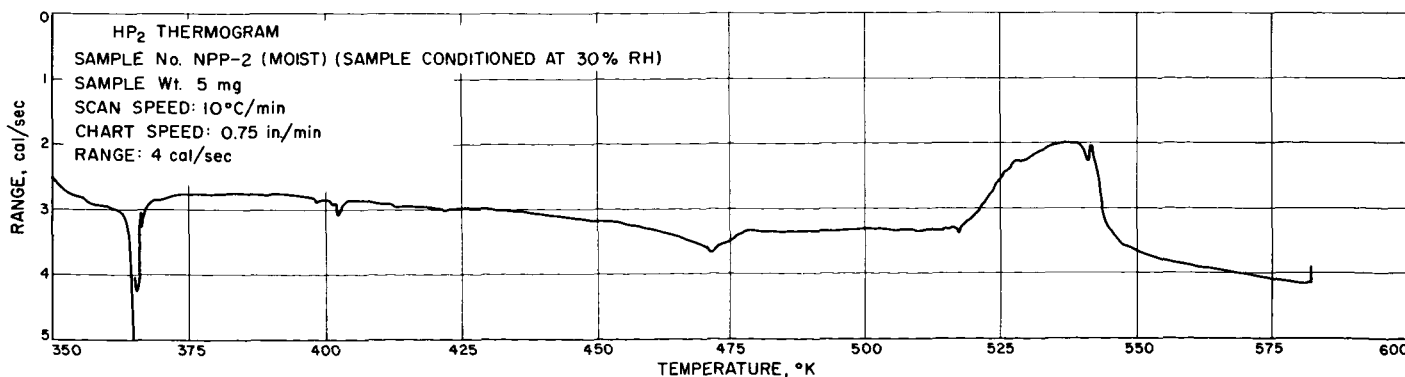


Fig. 3. Thermogram of moist  $\text{HP}_2$ , Sample NPP-2

atmosphere of 30% relative humidity. As can be noted, the crystalline phase change and the melting point are both exhibited at relatively the same temperatures as the dry sample which produced the thermogram in Fig. 2. However, the first exotherm did not occur following the melting of the sample. Only the final exotherm occurred, and that at a somewhat lower temperature than the dry sample, i.e., it peaked at approximately 265°C rather than just below 300°C. No attempt will be made at this time to explain these results. This effect of moisture is consistent from sample to sample, however, as is the decreased impact sensitivity. This study is continuing, and it is expected that a satisfactory explanation can be given shortly for the effects of moisture on HP<sub>2</sub>. Table 4 lists the phase change, melting point, and decomposition exotherm temperatures for samples from the three different lots of HP<sub>2</sub> being studied and also the results from some variations in the treatment of the samples.

### 5. Future Work

The studies reported here are being continued. Compatibility studies between HP<sub>2</sub> and potential binder ingredients have been started and will be continued, and a greater emphasis on formulation studies between HP<sub>2</sub> and a binder will be carried out.

## B. Applications Technology Satellite (ATS) Motor Development

R. G. Anderson

### 1. Introduction

In January 1963 the Jet Propulsion Laboratory initiated a development program to provide a solid propellant apogee rocket motor for a second generation SYNCOM satellite. This program, under the management of the Goddard Space Flight Center, was designated *Advanced SYNCOM*. It was to result in a spin-stabilized, active repeater communications satellite weighing about 750 lb, operating at synchronous altitude (22,300 miles) which would handle voice communications, teletype, and monochrome and color television signals.

In January 1964, the *Advanced SYNCOM* communication program was redirected to include a number of experimental instruments in addition to the original communication instruments. This expanded program is the *Applications Technology Satellite (ATS)* program and will result in a general-purpose satellite capable of operation at synchronous altitude with experimental instru-

Table 4. Thermal properties of hydrazine diperchlorate

Sample No.	Identification	Scan speed, °C/min	Phase change, °C	Melting point, °C	First exotherm		Final exotherm	
					Start °C	Peak °C	Start °C	Peak °C
1	TCC-1-1, dry	20	—	196	202	213	270	299
2	TCC-1-1, dry	5	—	—	Scan speed too low			
3	TCC-1-5, dry	10	—	196	201	206	252	297
4	TCC-1-1, dry	10	—	196	202	206	267	298
5 <sup>a</sup>	TCC-1-5, caked	10	—	—	Gradual decomposition, no peaks			
6	TCC-1-5, dry	10	88	196	201	206	252	299
7	NPP-2, dry	10	—	197	205	208	257	287
8 <sup>b</sup>	NPP-2, agg.	10	—	—	Gradual decomposition, no peaks			
9 <sup>c</sup>	NPP-2, CCl <sub>4</sub>	10	—	197	204	205	257	290
10	NPP-1, agg.	10	—	197	204	209	257	282
11 <sup>b</sup>	NPP-1, agg.	10	87	198	205	208	262	289
12 <sup>d</sup>	NPP-2, moist	10	92	197	NONE	NONE	245	265
13 <sup>e</sup>	TCC-1-1, dry	10	90	197	203	207	265	300
14	TCC-1-1, dry	10	88	196	203	219	242	278
15	TCC-1-1, dry	10	88	196	202	205	267	297
16	TCC-1-1, dry	10	87	196	202	217	252	300
17	TCC-1-1, dry	10	87	196	201	217	247	283

<sup>a</sup> Sample container had developed a leak admitting moist air which caused HP<sub>2</sub> to cake.

<sup>b</sup> Some lower density agglomerates screened out of sample.

<sup>c</sup> Sample with residual solvent present.

<sup>d</sup> Sample conditioned at a RH of 30%, Fig. 3.

<sup>e</sup> Fig. 2.



ments in the areas of meteorology, communications, radiation, navigation, gravity gradient stabilization, and various engineering experiments. For those satellites to be placed in synchronous orbit, JPL will provide a solid propellant rocket motor to provide the final required velocity increment at the apogee of the elliptical transfer orbit. This rocket motor is designated the JPL SR-28-1 (steel chamber) or JPL SR-28-3 (titanium chamber) rocket motor. It is presently intended that only the JPL SR-28-3 unit will be delivered for flight use.

Previous reports of progress on the development of this motor have been published in SPS 37-20 to 37-32, Vol. V.

## 2. Program Status Summary

The motor development program calls for static firing of four heavywall motors and 20 flightweight motors, including three with flight design titanium chambers prior to conducting a nine-motor qualification program. To date, the four heavywall motors plus 12 flightweight motors have been static-fired, two of which were under simulated high-altitude conditions at Arnold Engineering Development Center (AEDC), Tullahoma, Tennessee. All of the flightweight motors tested to date have been with type 410 chromium steel chambers.

During the period April-May 1965, two motors were shipped to AEDC for simulated high-altitude static testing. These units (D-3, D-4) are presently scheduled for testing during the week of May 23, 1965.

## 3. Environmental Test Program

The environmental test program consists of nine motor assemblies which will be subjected to various combinations of environments prior to static firing for confirmation of design integrity. The first seven motors will utilize steel chambers, and these motors have been cast. Three of the seven have been static tested. The last two of the nine units will utilize titanium chambers and will be subjected to all environments. A summary of the environmental program and current status is given in Table 5. Between each environment test, the motors are disassembled, thoroughly inspected, and the propellant grain subjected to radiographic inspection.

## 4. Nozzle Environmental Test

*a. Introduction.* The JPL-SR-28-1 solid propellant rocket motor is designed as the apogee kick motor to place the *Applications Technology Satellite (ATS)* into a synchronous orbit. Prior to the ignition of the apogee

Table 5. ATS apogee motor environmental test program summary

Test code	Motor number	Temperature cycling	Shipping temperature exposure	Centrifuge	Vibration	Spinning	Firing temperature °F	Current status, May 21, 1965
G-1	P-11	X	X	—	—	—	110	Static firing successful
G-2	P-10	X	X	—	—	—	10	Static firing successful
G-3	P-14	—	—	—	—	X	60	Static test—AEDC week of 5/23/65
G-4	P-15	—	—	—	X	X	60	Static test—AEDC week of 5/23/65
G-5	P-16	—	—	—	—	X	60	Static firing successful
G-6	P-19	X	—	X	X	X	60	Motor cast—environmental testing in process
G-7	P-20	X	—	X	X	X	10	Motor cast—environmental testing in process
G-8T	Titanium chamber	X	—	X	X	X	110	Chamber being fabricated
G-9T	Titanium chamber	X	—	X	X	X	10	Chamber being fabricated

motor, the spacecraft will complete three to five periods of an elliptical orbit with the apogee portion being at synchronous altitude. In traversing these elliptical orbits, the spacecraft will be eclipsed by the Earth during a portion of each orbit. During the eclipsed period, the nozzle exit cone of the apogee motor, which extends out of the main mass of the spacecraft, will be rapidly cooled thus establishing a nozzle exit cone temperature gradient (axial direction).

To evaluate the integrity of the nozzle exit cone under the above low-temperature conditions, a simulated low-temperature/low-pressure test was conducted at JPL on February 17, 18 and 19, 1965.

**b. Test article.** To measure nozzle temperature during the test phase, 38 thermocouples were attached to nozzle F-20 as shown in Fig. 4. Two identical sets of thermocouples were used to check for circumferential temperature gradients. Thermocouple sets are designated as N and Q series. The thermocoupled nozzle was then mated to an inert motor chamber P-6 so that actual heat transfer characteristics between nozzle and chamber could be

maintained. To prohibit the inert propellant and insulation from outgassing during low-pressure exposure the motor was sealed with an igniter closure at the head-end and an aluminum diaphragm over the nozzle entrance section. Two additional thermocouples were attached to the external surface of the motor chamber to monitor motor case and propellant temperatures during the temperature cycles. Ten layers of aluminum-coated Mylar were wrapped around the motor case (Fig. 5) in an effort to thermally isolate the motor chamber. In flight, the motor case will be submerged into the spacecraft. The assembled unit was then positioned in a head-end handling fixture and placed in a vertical, nozzle-end-up position in the environmental test chamber (Fig. 6).

**c. Environmental facilities and instrumentation.** A 6-ft-D environmental test chamber, located at JPL, was used for this test (Fig. 6). It consists of a large metal belljar type of chamber which is opened in a vertical direction and has low-pressure capabilities, and radiative temperature control. The large (approx 6 ft) radiative shield of the test chamber was not used for this test. A much smaller shield that covered only the nozzle exit

DUPLICATE SET OF THERMOCOUPLES PLACED 180 deg  
OPPOSITE SET SHOWN; NUMBERED Q1-Q20

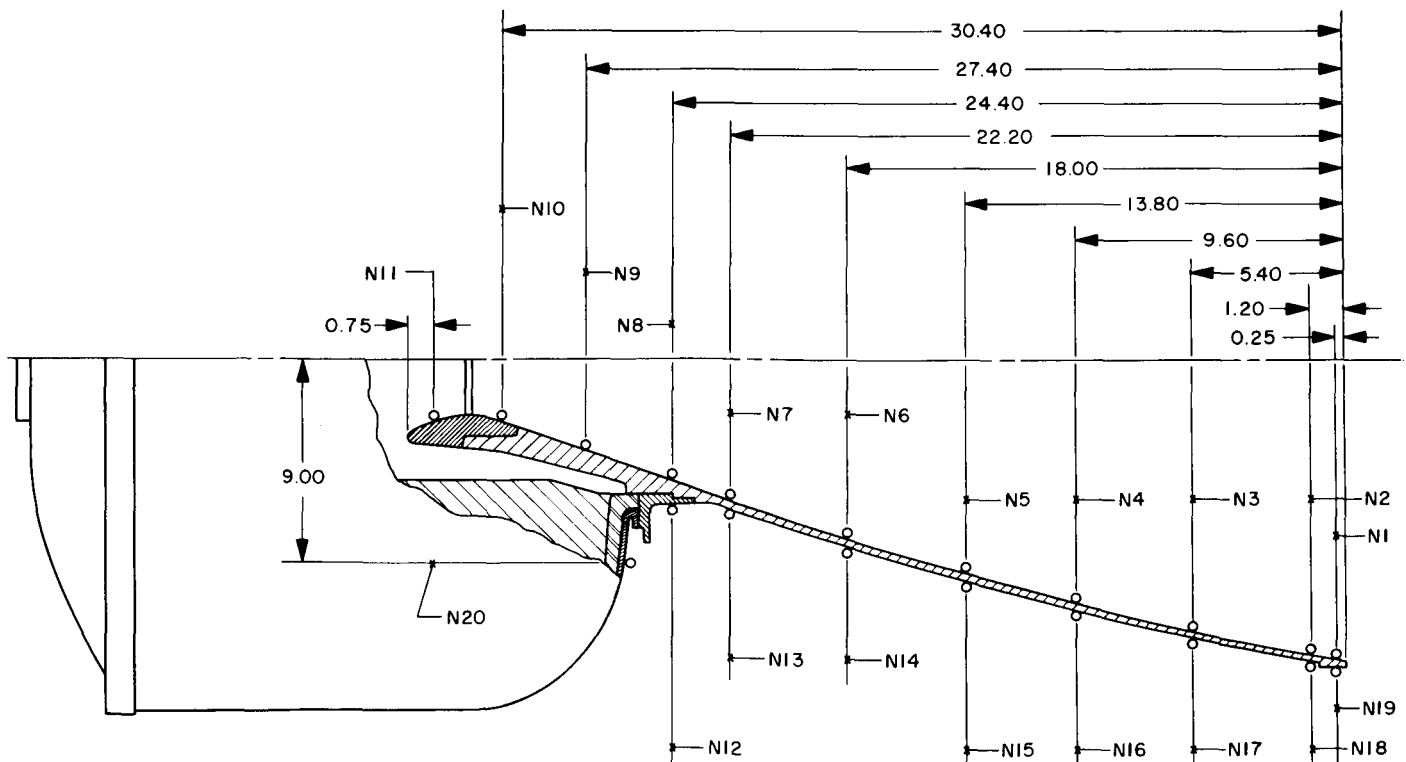


Fig. 4. Thermocouple placement for ATS nozzle environmental test

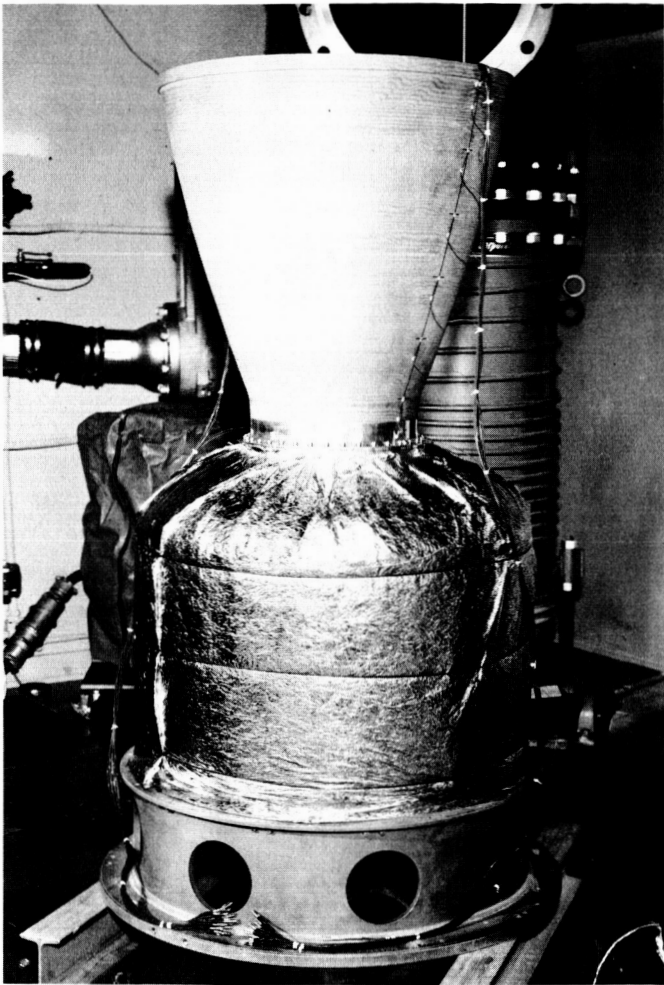


Fig. 5. ATS nozzle test article for environmental test

cone and provided a more rapid temperature response was employed. The radiative shield temperature is automatically controlled by the mass flow of  $\text{LN}_2$  or  $\text{GN}_2$ . In this test, maximum cooling capabilities of  $-300^\circ\text{F}$  were used.

The outputs from the 40 chromel-constantan thermocouples were recorded by a 50-channel Datex system. This system automatically records each thermocouple temperature at 5-min intervals. With the Datex temperature data, the cognizant engineer was able to determine the temperature gradients as they were established and could readily determine the precise times for temperature control change of the radiative shield.

*d. Test procedure.* After complete test article installation and instrumentation integrity checks, the large chamber was lowered and sealed. Vacuum pumping was initiated, and as the internal pressure reached  $3 \times 10^{-4}$

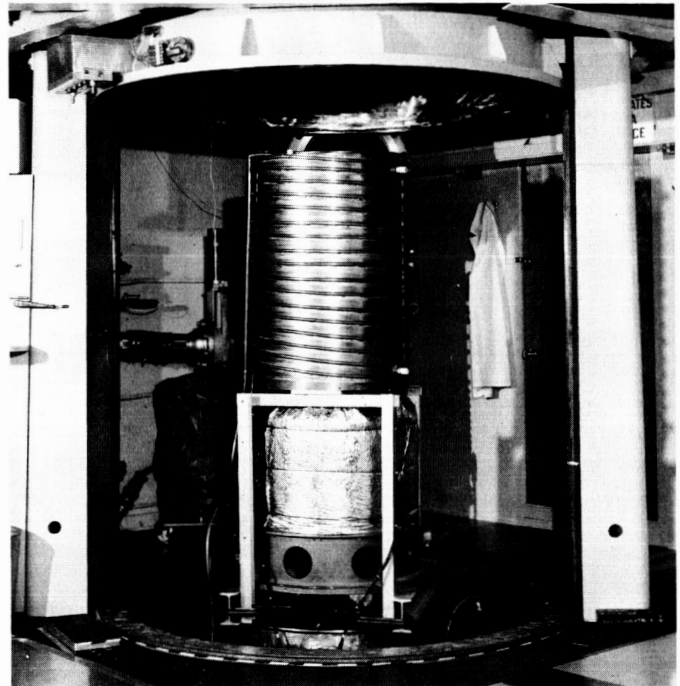


Fig. 6. Environmental test chamber with ATS nozzle test unit installed

mm of Hg,  $\text{LN}_2$  was forced through the radiative shield until an average shield temperature of  $-300^\circ\text{F}$  was established.

The purpose of the first cooling period was to ascertain the severity of temperature gradient that could be established and the duration of one complete temperature cycle. The low-temperature control point was thermocouple N-1, the cooling period cutoff point being when this thermocouple reached  $-190^\circ\text{F}$ . The cooling period was approximately 3 hr. After N-1 reached  $-190^\circ\text{F}$ , the radiative shield control temperature was changed to  $100^\circ\text{F}$  and maintained until nozzle ambient temperature conditions (approx  $70^\circ\text{F}$ ) were re-established. The heating period was approximately 1 hr. A total temperature cycle duration of 4 hr was required.

Following the completion of the first cycle, the bell chamber was raised and the nozzle was visually inspected for possible damage. No deleterious nozzle effects were observed due to the first temperature exposure. An inspection of the nozzle diaphragm revealed that the motor unit had lost some pressure during the initial, 4-hr cycle. A pressure transducer was installed to monitor motor chamber pressure during any additional testing.

After confirming nozzle integrity during the first cycle, a continuous 5-cycle test was initiated. This test subjected the nozzle to a similar temperature gradient as that established in the first cycle. Vacuum conditions were maintained for the 5 continuous cycles. The duration of this secondary test was 20 hr.

Upon completion of the 5-cycle test, the nozzle was removed from the chamber and put through a detailed visual and radiographic inspection.

*e. Results and discussion.* Fig. 7 shows the temperature values recorded by several thermocouples during the

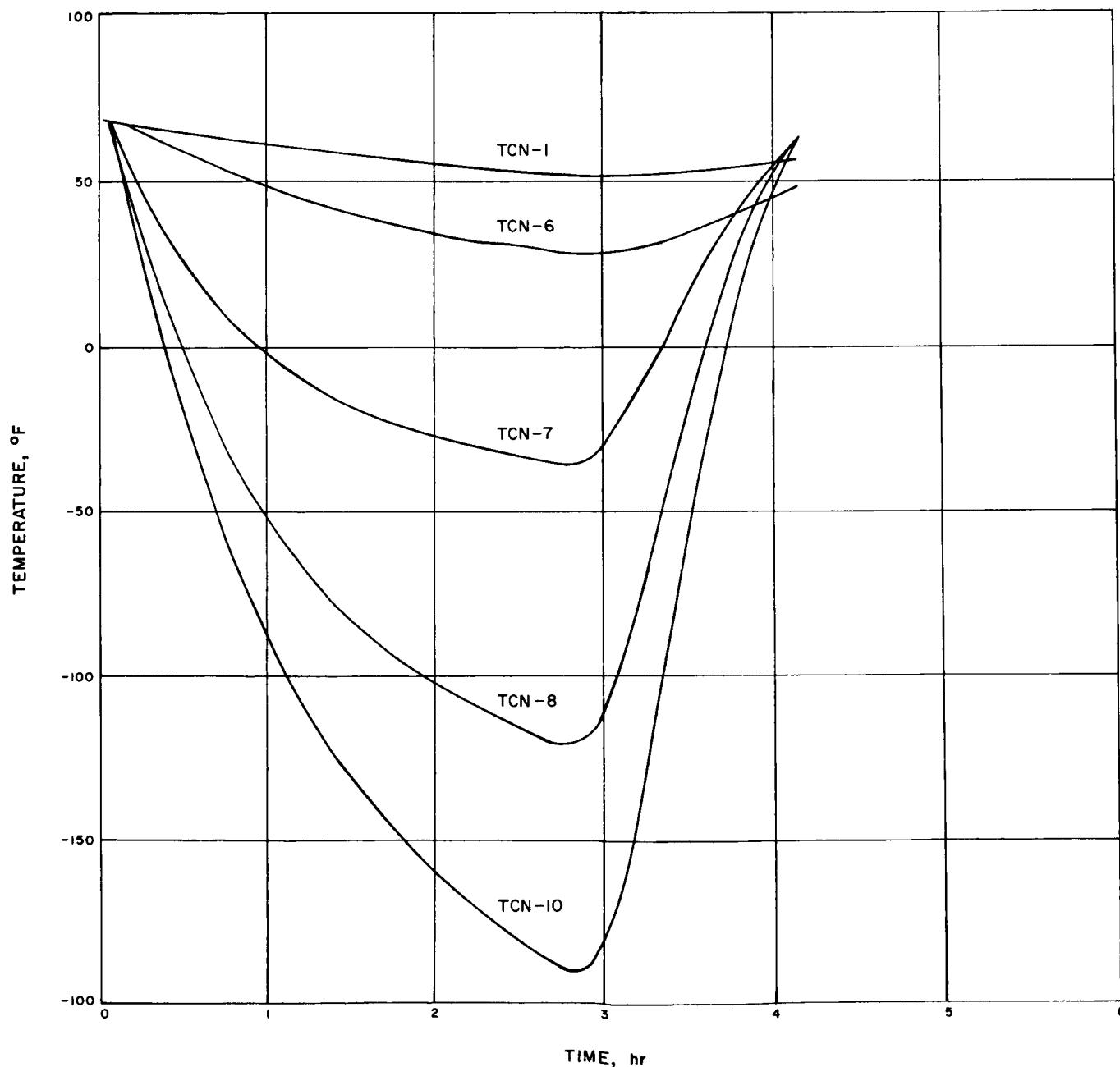


Fig. 7. Single-cycle ATS nozzle environmental test results

initial temperature cycle. These values are typical of any cycle. A portion of the temperature data recorded during the 5-cycle test is shown in Fig. 8. Fig. 9 shows nozzle temperature data as a function of specific nozzle locations. Several definite time periods, referenced to the start of nozzle cooling, have been plotted (Fig. 9) to show the established nozzle axial temperature gradients.

Visual and radiographic inspection of the nozzle revealed no deleterious effects due to the temperature/low-pressure environment. The test parameters (temperature and duration of exposure) are more severe than those expected during actual flight. Nozzle F-20 has been assigned to development test G-4 which will be static-fired under simulated altitude conditions at Arnold

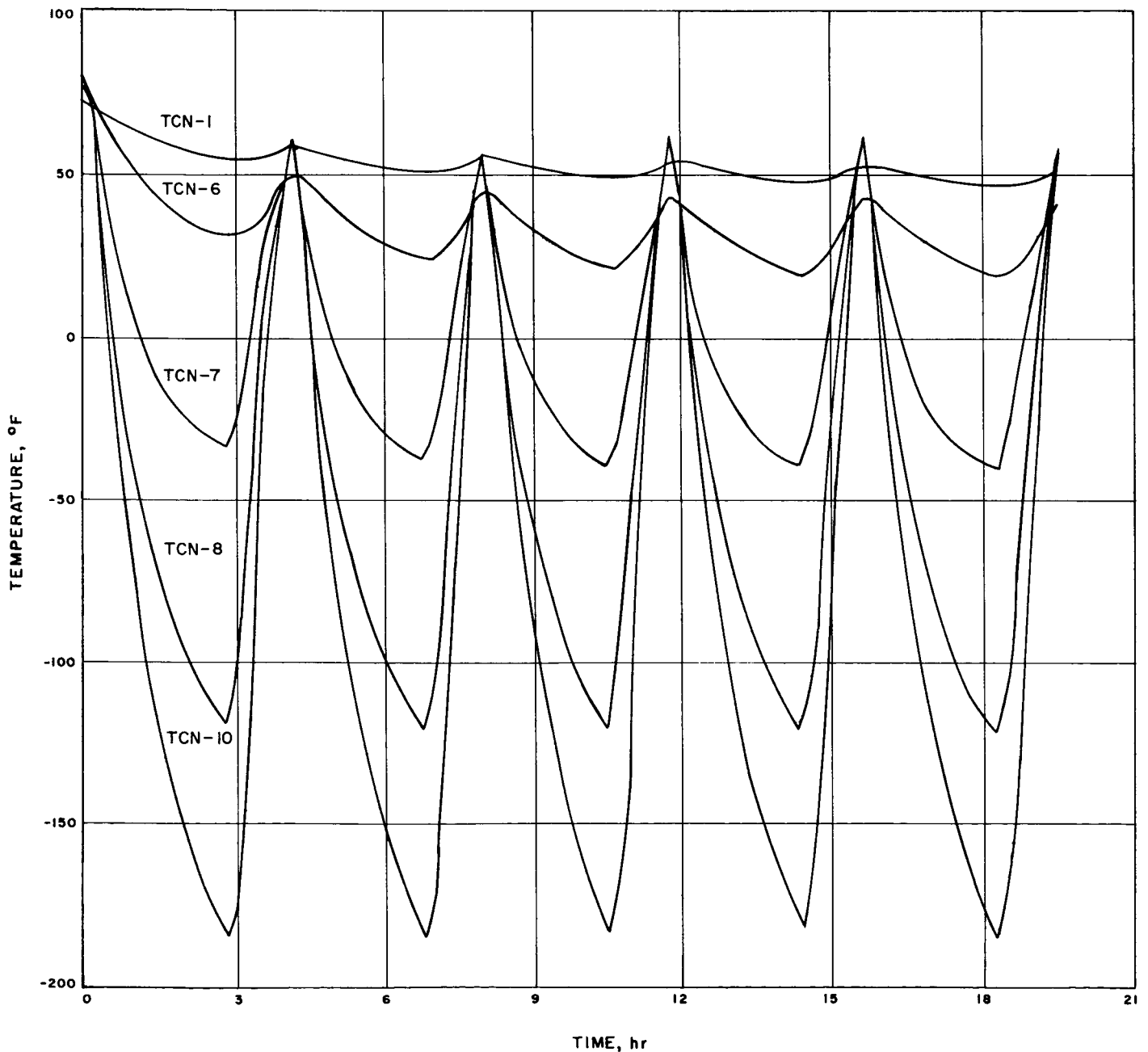


Fig. 8. Five-cycle ATS nozzle environmental test results

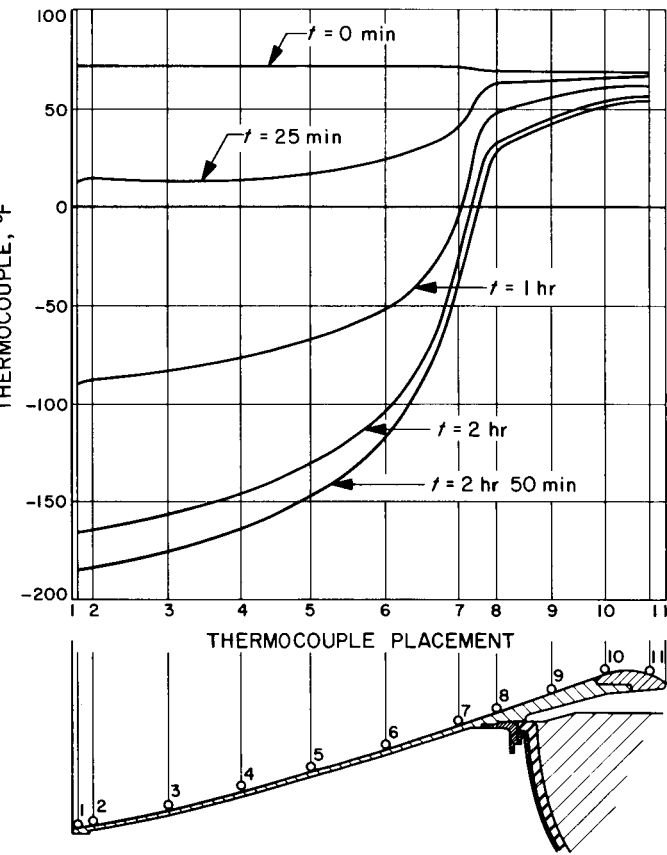
Engineering Development Center (AEDC) Tullahoma, Tennessee, during the week of May 23, 1965. This static test will confirm nozzle integrity after being subjected to severe temperature/low-pressure exposure.

**5. Motor Ignition Summary**

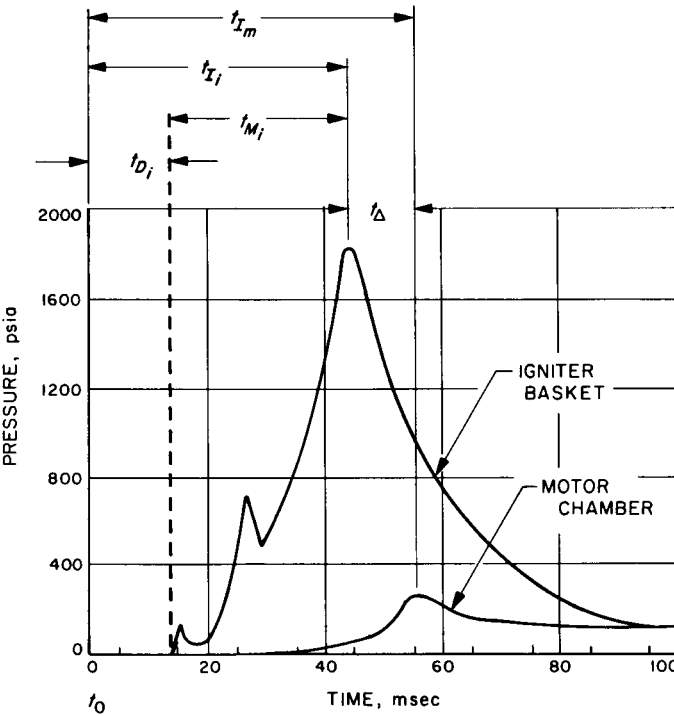
To date, a total of 16 static firings employing the ATS ignition system have been completed. Measured ignition

parameters have been defined (Fig. 10) and the ignition data are summarized in Table 6.

- $t_0$  = zero time, or time at which current is applied to squib
- $t_{Di}$  = the delay time from  $t_0$  until the first indication of squib pressure is seen
- $t_{Xi}$  = the time from  $t_0$  till peak igniter pressure is seen
- $t_{Xm}$  = the time from  $t_0$  till peak chamber ignition pressure is seen; this is not the same as peak run pressure
- $t_{\Delta}$  = the time difference between peak igniter basket pressure and peak chamber ignition pressure
- $t_{Mi}$  = the time from initial squib pressure till peak igniter basket pressure; the total igniter reaction time



**Fig. 9. ATS nozzle environmental test temperature profile**



**Fig. 10. Definition of ATS apogee motor ignition parameters**

Table 6. Igniter pressure-time data for all ATS motor ignition events<sup>a</sup>

Test	Test dated	Temp, °F	Vacuum start	$t_{D_i}$ , ms	$P_{I_i}$ , psia	$t_{I_i}$ , ms	$P_{I_m}$ , psia	$t_{I_m}$ , ms	$t_{\Delta}$ , ms	$t_{M_i}$ , ms
A-2	8/15/63	60	yes	14	1741	50	243	60	10	36
A-3	9/24/63	60	yes	8	2275	39	272	50	11	31
A-4	10/1/63	60	yes	13	2235	24	247	38	14	11
A-5	11/7/63	60	yes	12	2015	40	246	54	14	28
C-1	2/26/64	60	yes	11	1997	52	255	63	11	41
C-2	3/5/64	60	yes	11	1620	50	173	60	10	39
C-3	6/4/64	60	yes	21	1828	59	249	71	12	38
C-3A	6/12/64	60	yes	27	2289	48	270	63	15	21
C-4 <sup>b</sup>	7/14/64	10	yes	17	1853	34	228	45	11	17
E-1	7/16/64	60	yes	18	2060	58.5	224	68	9.5	40
C-5 <sup>b</sup>	7/23/64	60	yes	4	3292	21	273	29	8	17
E-2	10/13/64	60	yes	14	1832	37.5	233	49.2	11.7	23.7
H-1 <sup>b</sup>	11/4/64	10	yes	2.5	2005	20.5	224	40.5	20	18
G-5	11/24/64	60	no	10.0	1930	33	225	42	9	33
G-2 <sup>b</sup>	12/16/64	10	no	2.3	1527	21.5	212	32	10.5	19
G-1 <sup>b</sup>	12/16/64	110	(no oscillograph data)			—	—	—	—	—
		Mean average		14.4	1817	44.6	239.7	56	11.5	31

<sup>a</sup> Symbols are defined in Fig. 10.

<sup>b</sup> These tests were not used to derive the mean average.